

# PATENT SPECIFICATION

600,355



Convention Date (United States of America): Dec. 13, 1943.

Application Date (in United Kingdom): Sept. 26, 1944. No 18361/44.

Complete Specification Accepted: April 7, 1948.

Index at acceptance:—Classes 2(ii), A6; 15(ii), G(11: 12d: 21a1); and 140, E1a.

## COMPLETE SPECIFICATION

### Improvements in and relating to Treating Textile Materials

I, SIDNEY MILTON EDELSTEIN, a Citizen of the United States of America, of 11, Salem Park, Elizabeth, Union County, State of New Jersey, United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the treatment of textile material.

Certain cellulose ether solutions in alkali are at present in extensive use in the treatment of textile materials, to provide a coating thereon of cellulose ether. The dissolving of the cellulose ether in sodium hydroxide or like alkali metal hydroxide solution is effected with cooling, the extent of the cooling required depending in part on the degree of substitution of the alkoxy or ether groups in the cellulose molecule.

I have now discovered that alkyl ethers of cellulose may be dissolved in sodium zincate solution, without cooling to temperatures as low as required for dissolving a given ether in alkali metal hydroxide solution. Furthermore, the viscosity of solutions of alkyl ethers of cellulose in sodium zincate are substantially higher than the viscosities of comparable solutions in alkali metal hydroxide, in spite of the fact that I have found the zincate a much better solvent than the hydroxide for the said cellulose ethers. This increase of viscosity of the zincate solutions is desirable in decreasing the settling of pigments which are often suspended in the coating solution and thus facilitating the even and controlled application of colour by means of pigments to textile materials. Also, the use of zincate in place of alkali metal hydroxide solution as the medium for dissolving the said cellulose ethers permits of the use of ethers of lower degree of etherification than are possible when an alkali metal hydroxide solution is used as the solvent.

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In accordance with the invention a method is provided of treating textile material, which comprises applying thereto an aqueous solution of an alkali metal zincate having dissolved therein a cellulose alkyl ether and containing excess alkali metal hydroxide, precipitating the cellulose ether onto the material and removing a part but not all of the zinc from the precipitated alkyl cellulose ether by a washing treatment. Thus, amounts up to 1% of remaining zinc are not uncommon. Such retained zinc decreases the lustre when the textile material originally selected for the treatment is a rayon and increases the fastness to dyes and to pigments of textile materials that ordinarily are not satisfactory in fastness to these classes of colouring materials. Alkali treatment before washing increases the proportion of the zinc compound retained in the coating.

Fabrics treated with the zincate solutions of the cellulose alkyl ethers show more stiffness and greater fastness of the precipitated cellulose ether upon the fabric than is obtained when the ether is applied in an alkali metal hydroxide solution. In addition, the zincate used as the solvent medium has the power of swelling and dissolving the surface portions of textile material, especially rayon or other regenerated cellulose fabric, the material so dissolved being precipitated later, at the same time that the cellulose alkyl ether is precipitated from the adhering film of the zincate solution, so that the initially dissolved surface portions and the precipitated ether become firmly united in what amounts practically to an integral structure. As compared to solutions of cellulose in sodium zincate solution, the zincate solutions of the cellulose alkyl ether are more nearly clear and show less sediment on long standing with practically no tendency to set up to a gel on ageing.

In general, the method of making the

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solutions used in carrying out the method of the present invention comprises dispersing the selected cellulose alkyl ether in a solution of an alkali metal zincate and maintaining the dispersion until the cellulose ether is substantially completely dissolved, alkali metal hydroxide being in excess in the zincate.

It is convenient and preferred first to wet the cellulose alkyl ether with a portion of the water that is to be present in the final solution, as by slurring the cellulose alkyl ether with about 5 to 10 times its weight of water. In this preferred method, the solution of alkali metal zincate, of concentration in excess of that desired in the final solution, is then introduced, the whole stirred, and then the remainder of the water is added. The addition of the remainder of the water may be made gradually, in any convenient manner, as by flowing in the water in a small stream as the mixture is stirred or by the addition of the calculated quantity of cracked ice which, in melting, gives the water of dilution which is desired.

The ice if used effects cooling also.

It should be emphasized, however, that cooling is not required with the forms of cellulose alkyl ether that may be dissolved in alkali metal hydroxide solutions at temperatures of 0° C. or higher. With such cellulose ethers, good solutions are obtained at room temperatures, in summer or winter. With other cellulose alkyl ethers of lower degree of etherification, that is of lower degree of substitution of alkoxy groups in place of hydroxyls in the cellulose molecule, lower temperatures are helpful in effecting quick solution by my process. Thus, with cellulose alkyl ethers of degree of substitution requiring a temperature of -15° C. for satisfactory quick solution in aqueous sodium hydroxide, I use to advantage about 0° C. as the temperature for effecting solution in the zincate solution.

The time required to effect the solution is short. Ordinarily a few minutes stirring after the addition of the full amounts of all of the components of the finished solution is adequate to produce what appears to be and what remains a practically clear stable solution of the cellulose ether in the aqueous sodium zincate.

The proportions of water, alkali metal hydroxide, and zinc oxide used in making the sodium zincate solution constituting the final solvent medium for the cellulose ether may be 8 to 15 parts of sodium hydroxide, 2 parts to the saturation pro-

portion of zinc oxide, and 100 parts of water.

The proportion of the cellulose alkyl ether dissolved in the alkali metal zincate solution is ordinarily about 1 to 8 parts for 100 of total solution. For most uses the concentration and the viscosity of the finished solution are about right when the proportion of the cellulose ether is 3 to 6 parts for 100 parts of the solution.

The ether used is an alkali stable and preferably water insoluble cellulose alkyl ether as, for example, the methyl ether of degree of substitution less than 1.2, ethyl ether of d.s. less than 0.8, hydroxyethyl or other hydroxyalkyl ether of d.s. less than 1.3, carboxymethyl cellulose of d.s. less than 0.3.

The alkoxy content, the measure of the degree of etherification, is expressed in terms of the number of alkoxy groups substituted in the cellulose molecule for each glucose (C<sub>6</sub>) unit of the cellulose molecule and is called the degree of substitution sometimes abbreviated "d.s."

The zincate solvent makes possible the use of a wide range of ethers as classified by the degree of substitution and makes possible the convenient dissolving not only of alkyl ethers that are soluble in caustic alkalies but also of alkyl ethers of cellulose of such low degree of substitution down to 0.1 as to be impracticable for use commercially in making solutions of the ether in such alkalies.

In place of the water insoluble ethers, there may be used water soluble ethers such as the cellulose alkyl ethers of kind described, without any limitation as to the maximum degree of substitution. With such water soluble material, however, the advantages gained by the use of alkali metal zincate are not as great as with the water insoluble ethers.

The zincate which is used as the solvent medium is an aqueous solution of an alkali metal zincate. There is no advantage in using the more rare alkali metals in this composition. Either sodium or potassium zincate is satisfactory. The sodium zincate is preferred because of the lesser cost of sodium salts as compared to potassium salts.

Once the zincate solution of the cellulose ether has been applied to a textile material and the excess of solution has been removed, so as to leave an adhering film of the solution, then the cellulose ether is precipitated from the film upon the textile material. The precipitation may be effected by acidification, adding an electrolyte to give a high concentration of dissolved electrolyte, heating, or extreme dilution with water, alcohol, or other liquid that when mixed with a

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large proportion of water is a non-solvent for the cellulose ether.

After the textile is thus provided with a coating of precipitated cellulose ether, the product may be washed and dried. For some purposes the washing is omitted or purposely made imperfect. Also, the washing may be preceded by treatment with an aqueous solution of an alkali, of which a dilute solution of sodium carbonate, potassium carbonate, or sodium hydroxide is satisfactory.

The following specific Examples 1 to 5 illustrate the preparation of the zincate solution used in carrying out the method of the present invention.

#### EXAMPLE 1.

Six parts of hydroxyethyl cellulose ether of d.s. 0.5 is slurried with 20 parts of water until the ether is thoroughly wetted. To this slurry are added 22 parts of a sodium zincate solution containing 37% of actual sodium hydroxide and 13% of dissolved zinc oxide. The mix is then stirred thoroughly until the ether dissolves. The result is a thick solution. 52 parts of additional water are then stirred in, to make a total of 100 parts. The solution so made has a viscosity of about 1,000 centipoises.

#### EXAMPLE 2.

Five parts of the ether of Example 1 are slurried with 20 parts of water. Then this slurry is added to a zincate solution made with 22 parts of the above zincate solution of Example 1 and 53 parts of water. The whole mix is agitated for about 10 minutes. A solution similar to that of Example 1 is obtained.

#### EXAMPLE 3.

Five parts of hydroxyethyl ether of d.s. 0.1 is slurried with 20 parts of water and the mix is made as in Example 1 with the exception that the final 52 parts of water are replaced by an equal weight of ice, so that the whole mixture reaches a temperature of 0° C. The cellulose ether dissolves. Upon warming the solution to room temperature a stable solution of cellulose ether is obtained.

#### EXAMPLE 4.

Six parts of methyl ether of d.s. 0.2 is added to 100 parts of a solution containing 8 parts of caustic soda, 2.5 parts of dissolved zinc oxide and enough water to make 100 parts. The whole is mixed thoroughly at room temperature until the ether particles are thoroughly wet. The mass is then cooled to a temperature of 5° C. at which point the ether dissolves to give a homogeneous solution that

remains stable at this temperature indefinitely; or the solution may be warmed promptly to room temperature, at which also practically permanent stability is obtained.

#### EXAMPLE 5.

Any procedure of Examples 1 to 4 above is followed with the substitution, for the particular cellulose ether selected in those Examples, of equal proportions of any one or a mixture of the following ethers: ethyl, methyl, isopropyl, carboxymethyl, hydroxymethyl, or hydroxyethyl, each of d.s. 0.1 to 1.5 or more.

In order that the method of treating textile material in accordance with the invention may be fully understood, it will now be described with reference to the following Examples 6 to 9.

#### EXAMPLE 6.

A fabric is padded at room temperature through a zincate-ether solution prepared as described in any of the Examples 1 to 5 above. The fabric with adhering film of the solution is then passed through a bath containing an excess of a dilute solution of sulphuric acid in water, say 2 to 8% in concentration. The acid precipitates the cellulose ether upon the fabric. The product is then washed, the washing being discontinued short of removal of practically all the zinc and is combined with a subsequent alkali treatment, as with a solution of sodium carbonate, to neutralize remaining acid. This incomplete washing and alkali neutralization leaves in the fabric and coating a substantial total amount of zinc compound.

Finally the fabric is dried. The fabric may be cotton, regenerated cellulose or acetate rayon, a mixture of these materials containing also silk, or in some cases, even wool. It may be woven or knitted. In place of fabrics, yarns or threads may be used.

When a protein textile such as wool is used, the contact with the alkali metal zincate solution should be so brief that the fabric is not largely dissolved by the excess alkali present.

#### EXAMPLE 7.

The procedure of Example 6 is repeated except that, after passage through the acid, the fabric is not washed but passed through an aqueous sodium carbonate bath. Insoluble zinc carbonate is precipitated within the coated fabric. The product is then washed to remove the major part of the soluble salts but leave in the coated fabric a large proportion of the zinc compound.

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## EXAMPLE 8.

A cotton fabric or a yarn is padded through the zincate solution of the ether and is then dried in order to precipitate the cellulose ether. The fabric is then either washed thoroughly, to remove the alkali leaving a large part of the zinc behind as a basic zinc compound or hydroxide, or the fabric is soured in acid to remove the alkali and then washed and again dried.

## EXAMPLE 9.

The ether of Example 3 (d.s. 0.1) is used to treat a cotton fabric by the usual method of padding. After padding the ether-zincate solution, the solution on the fabric is then passed through a caustic solution of mercerizing strength (usually about 20 to 30% and preferably about 23%) but retaining some of the zinc and finished on any usual mercerizing machine and with usual technique, the cellulose ether being precipitated and fixed on the fabric at the time of passage through the mercerizing caustic solution. After the mercerization step the finishing is conventional. It includes washing on a tenter frame, souring and drying.

This method is applicable to any of the cellulose ethers of low degree of substitution as indicated by requiring a temperature substantially below room temperature for dissolving the ether in the zincate solution.

This treatment causes not only mercerization of the fabric but also of the cellulose ether coating. It gives an improved lustre of the finished coated fabric.

It will be understood that various modifications may be made in the Specification procedures described without

departing from the scope of the invention.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A method of treating textile material, which comprises applying thereto an aqueous solution of an alkali metal zincate having dissolved therein a cellulose alkyl ether and containing excess alkali metal hydroxide, precipitating the cellulose alkyl ether onto the material, and removing a part but not all of the zinc from the precipitated cellulose alkyl ether by a washing treatment.

2. A method according to claim 1, which includes subjecting the treated material to mercerization in strong alkali.

3. A method according to either of claims 1 or 2, in which the precipitation of the cellulose alkyl ether is effected by acidification.

4. A method according to any of claims 1 to 3, in which the textile material treated is regenerated cellulose, surface portions of which are dissolved in the solution during treatment and are subsequently precipitated along with the cellulose alkyl ether.

5. A method of treating textile material substantially as hereinbefore described with reference to Examples 6—9.

6. Treated textile material whenever obtained by the method according to any of claims 1 to 5.

Dated this 26th day of September, 1944.  
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